Catalytic Enantioselective Synthesis of Prostaglandin E1 Methyl Ester Using a Tandem 1,4-Addition-Aldol Reaction to a Cyclopenten-3,5-dione Monoacetal

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A Catalytic Asymmetric Synthesis of Prostaglandin E_1 Methyl Ester using a Tandem 1,4-Addition-Aldol Reaction to a Cyclopenten-3, 5-dione Monoacetal.

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Supporting information

General: Chromatography: silica gel Merk Typ 9385 230-400 mesh. TLC: silica gel 60, Merk, 0.25 mm. Optical rotations were measured on a Perkin-Elmer 241 MC (at RT). Mass spectra (HRMS) were obtained in an AEI MS-902. HPLC analysis was performed on a Water 600E system controller with a Waters 991 photodiode array detector. Enantiomere ratios were determined by chiral HPLC (DAICEL CHIRALPAK AD) in comparison with racemic material. ^{1}H-NMR and ^{13}C-NMR ($CDCl_3$): δ in ppm (δ = 7.24 ppm) for protons, (δ = 77 ppm) for carbon atoms were recorded on a Varian 200 MHz, 300 MHz or 500 MHz.
All reactions were carried out under argon atmosphere using dried glassware. Toluene, diethylether and THF were distilled from sodium and DCM, hexane, pentane and $CHCl_3$ was distilled from P_2O_5. All solvents were stored under nitrogen. BF_3*Et_2O (Aldrich) and triethylamine (Aldrich) was distilled before use. $Cu(OTf)_2$ (Aldrich) was dried before use. The following substances were commercial available and used without further purification: 2,2-dimethyl-1,3-propanediol (Aldrich), 2-cyclopenten-1,3-dione (Aldrich), PCC (Aldrich), 2-octyn-1-ol (Fluka), *t*-BuLi (1.7M pentane) (Aldrich), Red-Al® (3.4 M in toluene) (Aldrich), DMAP (Aldrich), chlorodimethylphenylsilane (Aldrich), oxalyl chloride (Aldrich), dimethyl sulfoxide (Aldrich). Bu_2Zn (1M heptane) (Aldrich) was distilled before use and diluted with toluene (1M solution).

General procedure for the acetalization of 2-cyclopenten-1,3-dione: To a cooled solution (0°C) of 2-cyclopenten-3,5-dione (1.92 g, 20 mmol) and BF_3*Et_2O (2.52 ml, 20 mmol) in 50ml chloroform was added the diol (1 eq).[1] After stirring for 3h the reaction mixture was poured into sat. NH_4Cl solution and extracted three times with 25 ml diethylether. The combined organic layers were dried over $MgSO_4$, filtered and concentrated in vacuo. Column chromatography SiO_2 (ether/pentane) gave the corresponding acetals.

8,8,dimethyl-6,10-dioxa-spiro[4.5]dec-3-en-2-one (1a): Purification by column chromatography SiO_2 (1:1 hexane/diethyl ether) Rf = 0.32 gave **1a**, 1.03g (28% yield) as white solid. ^{1}H-NMR (300 MHz) δ = 7.54 (d, J = 6Hz, 1H), 6.21 (d, J = 6Hz, 1H), 3.60 (d, J = 16Hz, 2H), 3.50 (d, J=16Hz, 2H), 2.60 (s, 2H), 1.09 (s, 3H), 0.89 (s, 3H); ^{13}C-NMR (200 MHz) δ= 204.1, 157.1, 135.3, 103.9, 72.9, 44.0, 29.9, 22.3, 22.2; HRMS calcd for $C_{10}H_{14}O_3$ 182.094, found 182.094; Anal.Caldc. for $C_{10}H_{14}O_3$: C, 65.90; H, 7.74 found C, 65,98; H, 7.79.

8,8,diphenyl-6,10-dioxa-spiro[4.5]dec-3-en-2-one (1b): Purification by column chromatography SiO_2 (1:1 hexane/diethyl ether) Rf = 0.43 gave **1b** 3.06g (50% yield) as white solid. ^{1}H-NMR (300 MHz) δ = 7.51 (d, J = 6 Hz, 1H), 7.41-7.20 (m, 10H), 6.21 (d, J = 6 Hz, 1H), 4.65 (d, J = 11.7 Hz, 2H), 4.35 (d, J = 11.7 Hz, 2H), 2.70 (s, 2H); ^{13}C-NMR (200 MHz) δ = 203.3, 156.5, 143.4, 143.2, 135.1, 128.4, 128.2, 127.6, 126.7, 126.7, 126.4, 104.0, 70.2, 44.4, 44.0; MS *m/z* 324 ($M + NH_4^+$).

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[1] For the synthesis of 2,2-diphenylpropane-1,3-diol: Markees, D. G.; Burger, A. *J. Am. Chem. Soc.* **1949**, *71*, 2031.

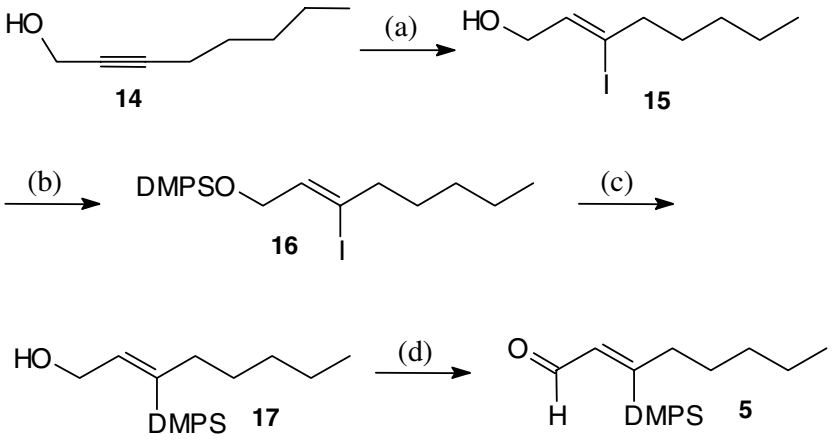
(General) procedure for the tandem 1,4-addition-aldol reaction: A solution of $Cu(OTf)_2$ (3.6 mg, 0.01 mmol) and phosphoramidite **4**[2] (10.7 mg, 0.02 mmol) in 7 ml toluene was stirred under a nitrogen atmosphere at ambient temperature for 1 h. 8,8-dialkyl-6,10-dioxaspiro[4.5]dec-3-en-2-one **1b** (0.5 mmol) and p-bromobenzaldehyde (92.5 mg, 0.5 mmol) was added. After cooling the reaction mixture to -45 °C, dibutylzinc 0.5 ml (1M solution in toluene) was added and stirring at -45°C was continued for 18 hours. After complete conversion, the reaction mixture was poured in 25 ml of NH_4Cl (aq) and the organic layer was separated, and the aqueous layer was extracted with diethyl ether two times. The combined organic layers were dried over $MgSO_4$, filtered and concentrated in vacuo.

3-[(4-bromophenyl)(hydroxy)methyl]-4-butyl-8,8-diphenyl-6,10-dioxaspiro[4.5]decan-2-one (2f). Purification by column chromatography SiO_2 (2:1 pentane/diethyl ether) Rf = 0.28 gave **2f**, 174 mg (64% yield) as colorless oil which solidified upon standing. ^{1}H-NMR (300 MHz) δ = 7.42- 7.05 (m, 14H), 4.75 (dd, J = 7.8 Hz, J = 1.8 Hz, 1H), 4.53-4.46 (m, 2H), 4.31-4.16 (m, 2H), 3.84 (d, J = 1.8 Hz, 1H (OH)), 3.03 (dd, J = 18 Hz, J = 1.8 Hz, 1H), 2.53 (d, J = 18.3 Hz, 1H), 2.39 (t, J = 7.5 Hz, 1H), 2.02 (qa, J = 7.2 Hz, 1H), 1.40 (m, 1H), 0.84- 0.69 (m, 5H), 0.57 (t, J = 7.2 Hz, 3H); ^{13}C-NMR (300 MHz) δ = 215.9, 143.3, 143.1, 140.1, 131.4, 128.7, 128.6, 128.3, 127.9, 126.9, 126.6, 126.4, 121.9, 103.9, 74.4, 70.1, 68.9, 59.6, 47.0, 45.2, 44.8, 29.6, 27.6, 22.7, 13.7. EI^+ (550), (364)

(General) procedure for the oxidation to a diketone: To **2f** (150 mg, 0.27 mmol) in CH_2Cl_2 (5 ml) were added molecular sieves (4 Å, 0.5 g) and PCC (215 mg, 1 mmol) at 0°C. The reaction mixture was stirred for 4h at room temperature and diluted with diethyl ether, filtered over celite and evaporated to dryness.

3-(4-bromobenzoyl)-4-butyl-8,8-diphenyl-6,10-dioxaspiro[4.5]decan-2-one (3f). Purification by column chromatography SiO_2 (5:1 pentane/diethyl ether) Rf = 0.30 gave **3f**, 91.7 mg (62% yield) as colorless oil which solidified upon standing. ^{1}H-NMR (300 MHz) δ=7.82-7.02 (m, 14H), 4.62 (d, J = 11.7 Hz, 2H), 4.41-4.13 (m, 2H), 3.33 (d, J = 17.7 Hz, 1H), 3.10 (m, 1H), 2.48 (d, J = 17.7 Hz, 1H), 1.51 (m, 1H), 1.22-0.74 (m, 5H), 0.62 (t, J = 6.9 Hz, 3H); ^{13}C-NMR (200 MHz) δ= 206.3, 193.5, 143.2, 142.9, 135.6, 131.9, 130.9, 128.7, 128.6, 128.1, 127.1, 126.3, 126.2, 102.9, 71.4, 68.6, 62.7, 48.8, 44.9, 44.7, 30.2, 27.0, 22.8, 13.6; HRMS calcd for $C_{31}H_{31}O_4Br$ 548.138 found 548.139; The e.e. of 97 % was determined by HPLC on a chiral stationary phase (DAICEL CHIRALPAK AD, iPrOH:hexane 50:50, flow: 1ml/min, RT, T_r = 5.70 min, T_r = 9.43)

Synthesis of (5)[a]:



[a]Key: (a) (1) Red-Al[@], THF, 5h; (2) I_2, -78ºC; (b) DMPSCl, Et_3N, DMAP, CH_2Cl_2, 0ºC
(c) *t*-BuLi 2 equ., THF, -78ºC; (d) $(COCl)_2$, Me_2SO, Et_3N, -78ºC.

[2] Feringa, B.L.; Pineschi, M.; Arnold, L.A.; Imbos, R.; de Vries, A.H.M. *Angew. Chem. Int. Ed. Engl.* **1997**, *36*, 2620.

(Z)-3-Iodo-2-octen-1-ol (15). Under argon atmosphere, Red-Al (3.4 M in toluene, 49 g, 338 mmol) was dissolved in diethyl ether (600 ml). To this mechanical stirred solution maintained at 0 ºC was added 2-octyn-1-ol **14** (25 ml, 169 mmol) in ether (50 ml), dropwise. After 4h at room temperature, the reaction mixture was re-cooled to 0 ºC and quenched by addition of ethyl acetate (16.5 ml, 169 mmol). After the mixture was cooled to –78 ºC, iodine (64g, 254 mmol) was added in one portion and the reaction mixture was allowed to warm up to room temperature over 2h. The reaction mixture was quenched by slow addition of saturated Na_2SO_3 (aq), and the organic layer was separated and washed with Na_2SO_3 (aq), water and saturated NaCl (aq). The resulting organic solution was dried over $MgSO_4$, filtered and concentrated in vacuo. Column chromatography SiO_2 (1:6 ether/pentane) gave 38.8g (94%) of **15**, as light purple colored oil: ^{1}H-NMR (300 MHz) δ = 5.78 (tri, J = 5.9 Hz, 1H), 4.14 (d, J = 5.9 Hz, 2H), 2.44 (tri, J =7.4 Hz, 2H), 1.48 (qui, J = 7 Hz, 2H), 1.24 (m, 4H), 0.84 (tri, J = 6.6 Hz, 3H); ^{13}C-NMR (200 MHz) δ = 133.2, 111.0, 67.3, 45.1, 30.4, 28.8, 22.4, 14.0; CI$^+$ 254, 272 (NH_4^+)

(Z)-1-dimethyl(phenyl)siloxy-3-Iodo-2-octene (16). To a solution of **15** (14g, 59 mmol) in CH_2Cl_2 (300 ml) was added Et_3N (8.3 ml, 60 mmol) and a catalytic amount of DMAP. At 0°C chlorodimethylphenylsilane (10 ml, 59 mmol), in CH_2Cl_2 (20 ml) was added over a period of 1 hour. After stirring for an additional 1 h, the reaction mixture was poured into a NH_4Cl (aq) and the organic layer was separated, dried with $MgSO_4$, filtered and concentrated in vacuo. The residue was resolved in pentane (300 ml) and washed with water, NaCl (aq) and dried over $MgSO_4$ filtered and concentrated in vacuo. The colorless oil 20.6 g (92 %) was used without further purification. ^{1}H-NMR: 7.60-7.50 (m, 2H), 7.40-7.32 (m, 3H), 5.71 (tri, J = 5.4 Hz, 1H), 4.17 (d, J = 5.4 Hz, 2H), 2.39 (tri, J = 6.8 Hz, 2H), 1.45 (qui, J = 6.6 Hz, 2H), 1.24 (m, 6H), 0.87 (tri, J = 6.5 Hz, 3H), 0.39 (s, 6H); ^{13}C-NMR (200 MHz) δ = 142.3, 133.9, 133.4, 129.7, 127.8, 108.4, 68.2, 45.0, 31.9, 30.4, 22.4, 14.0, -1.7; CI$^+$ 406 (NH_4^+)

(*Z*)-3-dimethyl(phenyl)silyl-2-octen-1-ol (17). Under argon atmosphere **16** (30 g, 77 mmol) was dissolved in THF (400 ml). At –78°C, 2.2 equivalents of *t*-BuLi (100ml, 1.7 M pentane) was added dropwise over a periode of 10 min and stirred for 2 hours at the same temperature. The reaction mixture was quenched with NH_4Cl (aq) (250 ml), the organic layer was separated, and the aqueous layer was extracted twice with 100 ml diethyl ether. The combined organic layers were dried over $MgSO_4$, filtered and concentrated in vacuo. Column chromatography SiO_2 (1:6 diethyl ether/pentane) gave 14.8 g (74%) of **17**, as colorless oil: Rf = 0.35; ^{1}H-NMR (300 MHz) δ = 7.54-7.50 (m, 2H), 7.37-7.28 (m, 3H), 6.21 (tri, J = 5.7 Hz, 1H), 3.94 (d, J = 5.7 Hz, 2H), 2.19 (tri, J = 6.9 Hz, 2H), 1.41-1.19 (m, 8H), 0.88 (tri, J = 6.8 Hz, 3H), 0.40 (s, 6H); ^{13}C-NMR (200 MHz) δ = 142.4, 141.4, 139.5, 133.5, 129.0, 128.0, 62.3, 38.3, 31.9, 30.1, 22.5, 14.0, -1.0; CI$^+$ 262, 280 (NH_4^+).

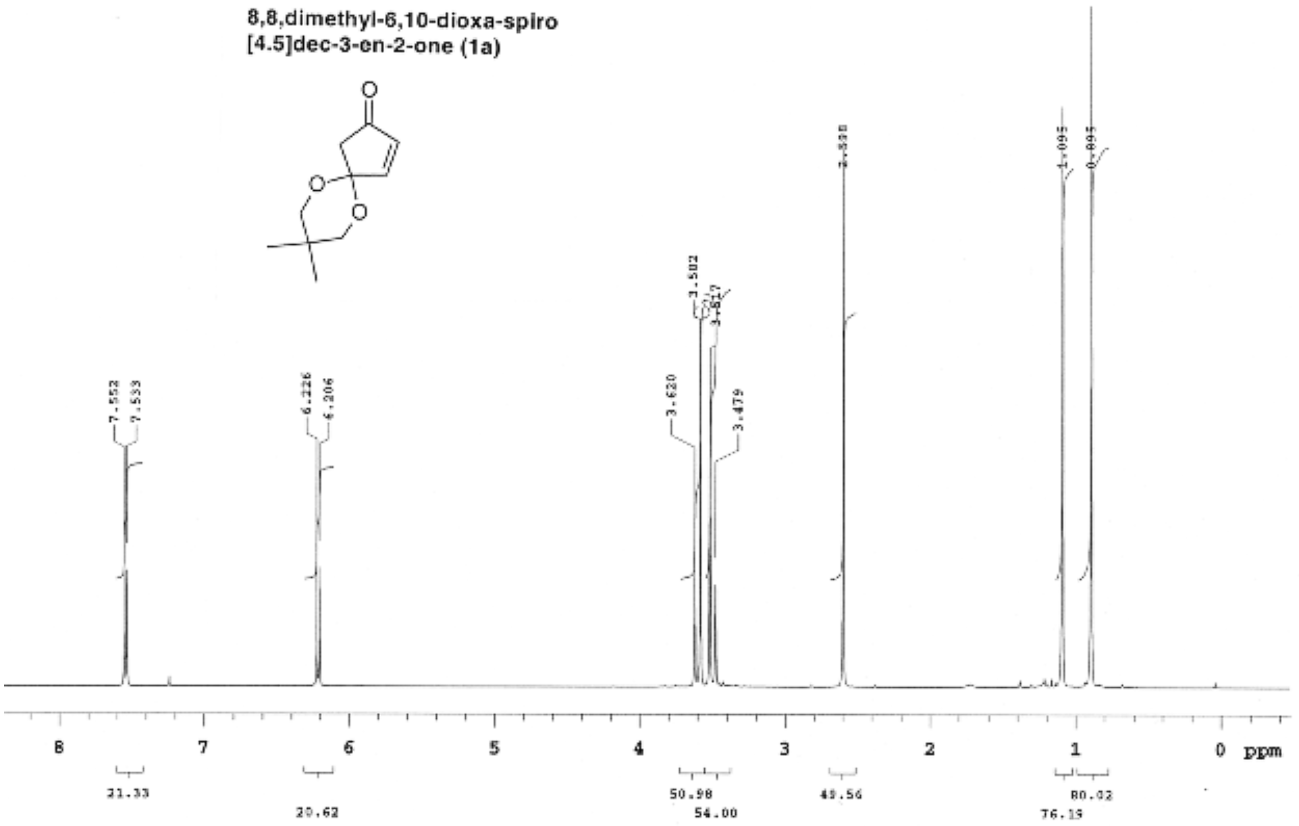
(Z)-3-dimethyl(phenyl)sily-2-octenal (5). To a solution of $(COCl)_2$ (3.7 ml, 42 mmol) in THF (20 ml) was added Me_2SO (6 ml, 84 mmol) in CH_2Cl_2 (10 ml) at –78° C and the resulting solution was stirred for 5 min. To this solution alcohol **17** (10g, 38 mmol) in THF (20 ml) was added slowly and the mixture was stirred for 30 min, followed by the addition of Et_3N (26ml, 190 mmol) in CH_2Cl_2 (300 ml). The reaction mixture was warmed up to room temperature and quenched with NH_4Cl (aq) and the organic layer was separated, and the aqueous layer was extracted with diethyl ether two times. The combined organic layers were dried over $MgSO_4$, filtered and concentrated in vacuo. Ratio of *(Z:E)* was 93:7. Flash column chromatography SiO_2 (1:50 ether/pentane) gave 7.8 g (79 %) of **5**, as bright yellow oil: Rf = 0.34; 100% (Z); ^{1}H-NMR (300 MHz) δ = 9.68 (d, J = 8.5 Hz, 1H), 7.50-7.43 (m, 2H), 7.35-7.30 (m, 3H), 6.45 (d, J = 8.5 Hz, 1H), 2.33 (tri, J = 8 Hz, 2H), 1.35-1.20 (m, 6H), 0.83 (tri, J = 6.7 Hz, 3H), 0.50 (s, 6H); ^{13}C-NMR (200 MHz) δ = 192.8, 170.8, 141.5, 138.9, 133.4, 129.4, 128.0, 39.0, 31.3, 28.6, 22.2, 13.7, -0.70; CI$^+$ 278 (NH_4^+).

Methyl 7-(2-{(*Z*)-3-[dimethyl(phenyl)silyl]-1-hydroxy-2-octenyl}-3-hydroxy-8,8-diphenyl-6,10-dioxaspiro[4.5]dec-1-yl)heptanoate (8). Under argon atmosphere, a solution of **7**[3] in diethyl ether (40 ml) was treated with $Zn(BH_4)_2$[4] (20 ml, 0.5 M in diethyl ether) at –30°C. After stirring for 3 h at the same temperature the reaction mixture was quenched with NH_4Cl (aq) (250 ml) in a beaker and stirred for 30 min. The reaction mixture was diluted with diethyl ether (100 ml) and the organic layer was separated. The aqueous layer was extracted two times with diethyl ether and the combined organic layers were dried over $MgSO_4$, filtered and concentrated in vacuo. Column chromatography SiO_2 (5:4 ether/pentane) gave **8,** 807 mg (38% yield over two steps) as colorless oil: Rf = 0.30; ^{1}H-NMR (300 MHz) δ = 7.47-6.99 (m, 15H), 6.03 (d, J = 10 Hz, 1H), 4.53-4.42 (m, 2H), 4.31-4.24 (m, 2H), 3.94 (m, 2H), 3.63 (s, 3H), 2.44 (d, J = 5.6Hz, 1H) OH, 2.23-2.04 (m, 6H), 1.57-0.80 (m, 22H), 0.38 (s, 3H), 0.33 (s, 3H); ^{13}C-NMR (200MHz) δ = 174.3, 144.0, 143.7, 143.6, 143.2, 139.8, 133.4, 129.2, 128.6, 128.5, 128.1, 128.0, 126.8, 126.4, 126.1, 107.3, 72.9, 72.1, 70.0, 68.7, 56.8, 51.4, 49.2, 44.8, 38.4, 37.8, 34.1, 31.8, 30.2, 29.8, 29.0, 28.6, 27.9, 25.0, 22.5, 14.0, -0.9, -1.0; EI^+ 712, 696, 664, 633, 501; the ee of 93.6 % was determined by HPLC on a chiral stationary phase (DAICEL CHIRALPAK AD, iPrOH:heptane 25:75, flow: 1 ml/min, RT, T_r = 4.9 min, T_r = 9.0), $[\alpha]_D^{23}$-31° (c 0.9, $CHCl_3$),

Prostaglandin E_1 methyl ester (13) (characterisation). ^{1}H-NMR (500 MHz) δ = 5.64 (dd, J = 15.0 Hz, J = 7.3 Hz, 1H), 5.52 (dd, J = 15.0 Hz, J = 8.7 Hz, 1H), 4.08 (m, 1H), 4.01 (m, 1H), 3.63 (s, 3H), 2.70 (dd, J = 18.6 Hz, J = 7.3 Hz, 1H), 2.32 (dt, J = 12.1 Hz, J = 8.8 Hz, 1H), 2.26 (t, J = 7.7 Hz, 2H), 2.20 (dd, J = 18.3 Hz, J = 9.9 Hz, 1H), 1.96 (dt, J = 12.1 Hz, J = 5.9 Hz, 1H), 1.60-1.43 (m, 6H), 1.36-1.24 (m, 12H), 0.8 (t, J = 6.9 Hz, 3H); ^{13}C-NMR (300MHz) δ = 214.6, 174.3, 136.8, 131.8, 73.0, 71.8, 54.8, 54.4, 51.5, 45.8, 37.3, 34.0, 31.6, 29.3, 28.8, 27.6, 26.6, 25.1, 24.8, 22.6, 14.0; $[\alpha]_D^{23}$-51° (c 1.0, CH_3OH),

[3] Compound **7** was synthesized using the general procedure for the tandem reaction on a 3 mmol scale and used without further purification.

[4] Compound was synthezised according: Gensler, W. J.; Johnson, F. A.; Sloan, A. D. *J. Am. Chem. Soc.* **1960**, *82*, 6078.


8,8,dimethyl-6,10-dioxa-spiro
[4.5]dec-3-en-2-one (1a)
O
O
O
7.552
7.533
6.226
6.206
3.620
3.502
3.479
2.538
1.095
0.893
8
7
6
5
4
3
2
1
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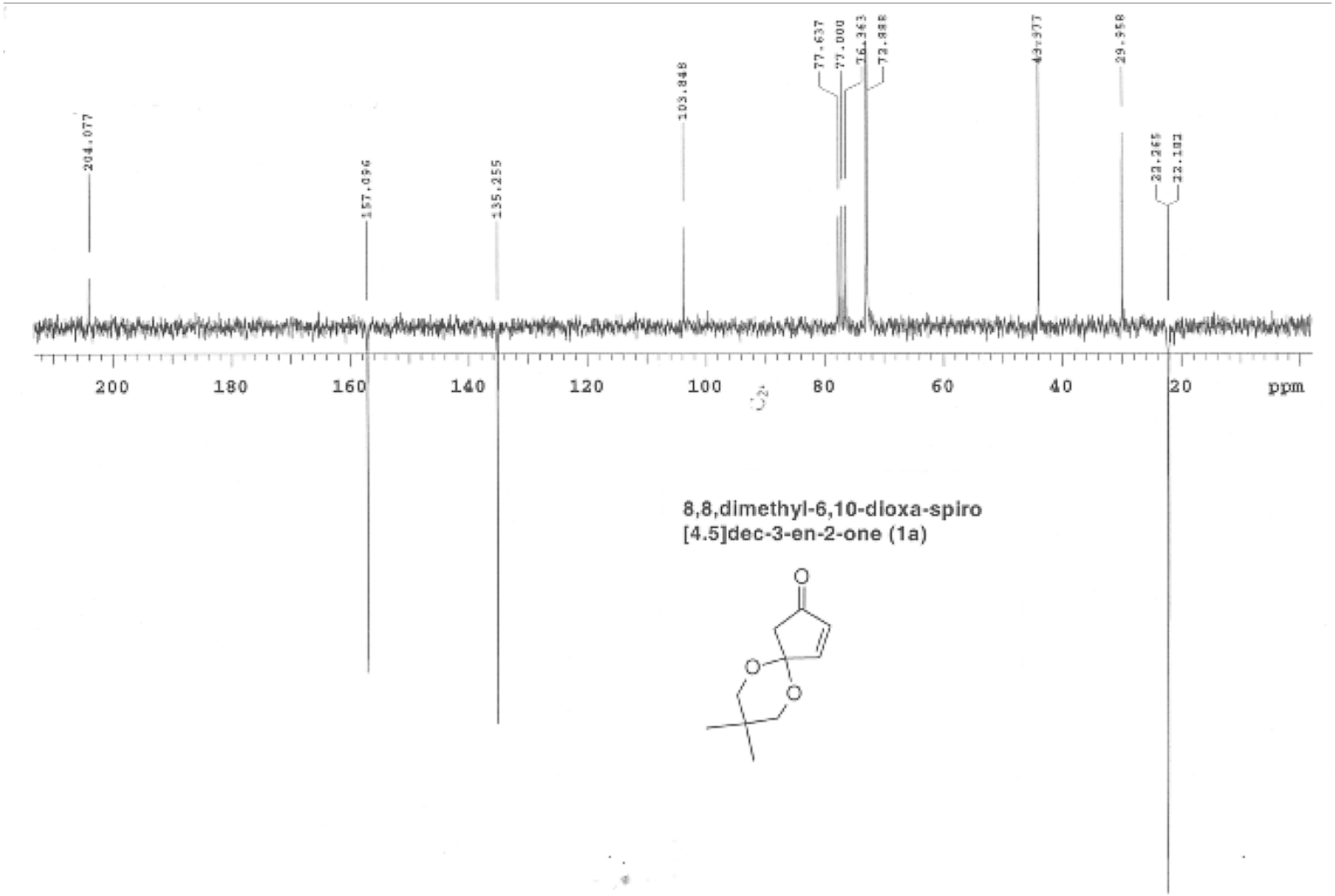
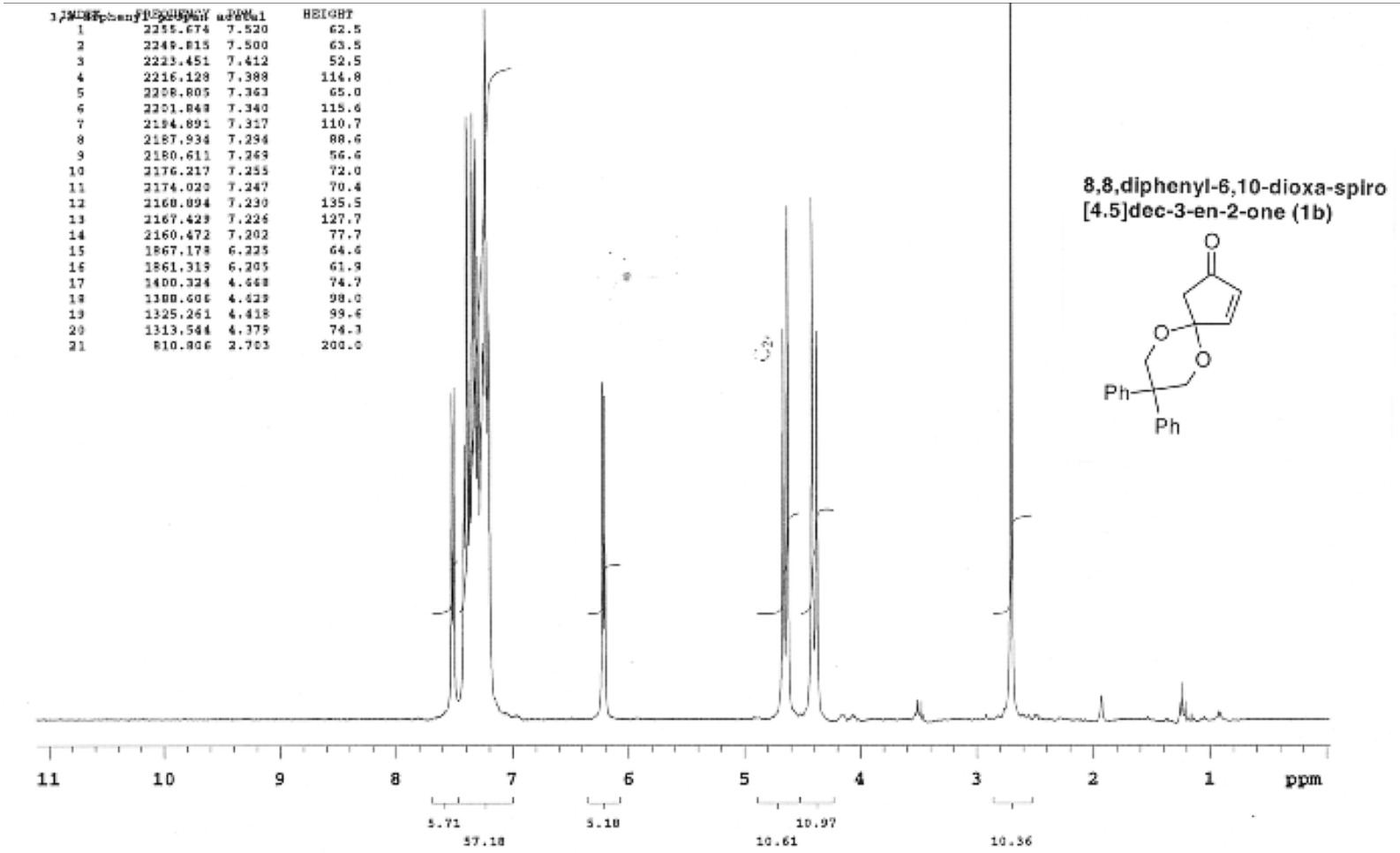
80.02



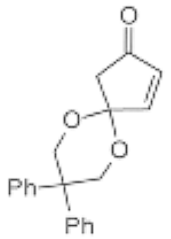
8,8,dimethyl-6,10-dioxa-spiro
[4.5]dec-3-en-2-one (1a)



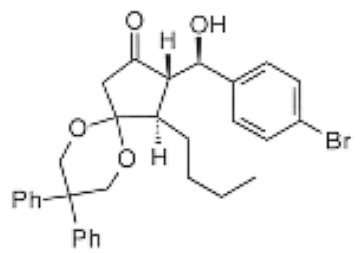
	FREQUENCY	PPM	HEIGHT
1	2255.674	7.520	62.5
2	2249.815	7.500	63.5
3	2223.451	7.412	52.5
4	2216.128	7.388	114.8
5	2208.805	7.363	65.0
6	2201.848	7.340	115.6
7	2194.891	7.317	110.7
8	2187.934	7.294	88.6
9	2180.611	7.269	56.6
10	2176.217	7.255	72.0
11	2174.020	7.247	70.4
12	2168.094	7.230	135.5
13	2167.429	7.226	127.7
14	2160.472	7.202	77.7
15	1867.178	6.225	64.6
16	1861.319	6.205	61.9
17	1400.324	4.668	74.7
18	1388.606	4.629	98.0
19	1325.261	4.418	99.6
20	1313.544	4.379	74.3
21	810.806	2.703	200.0

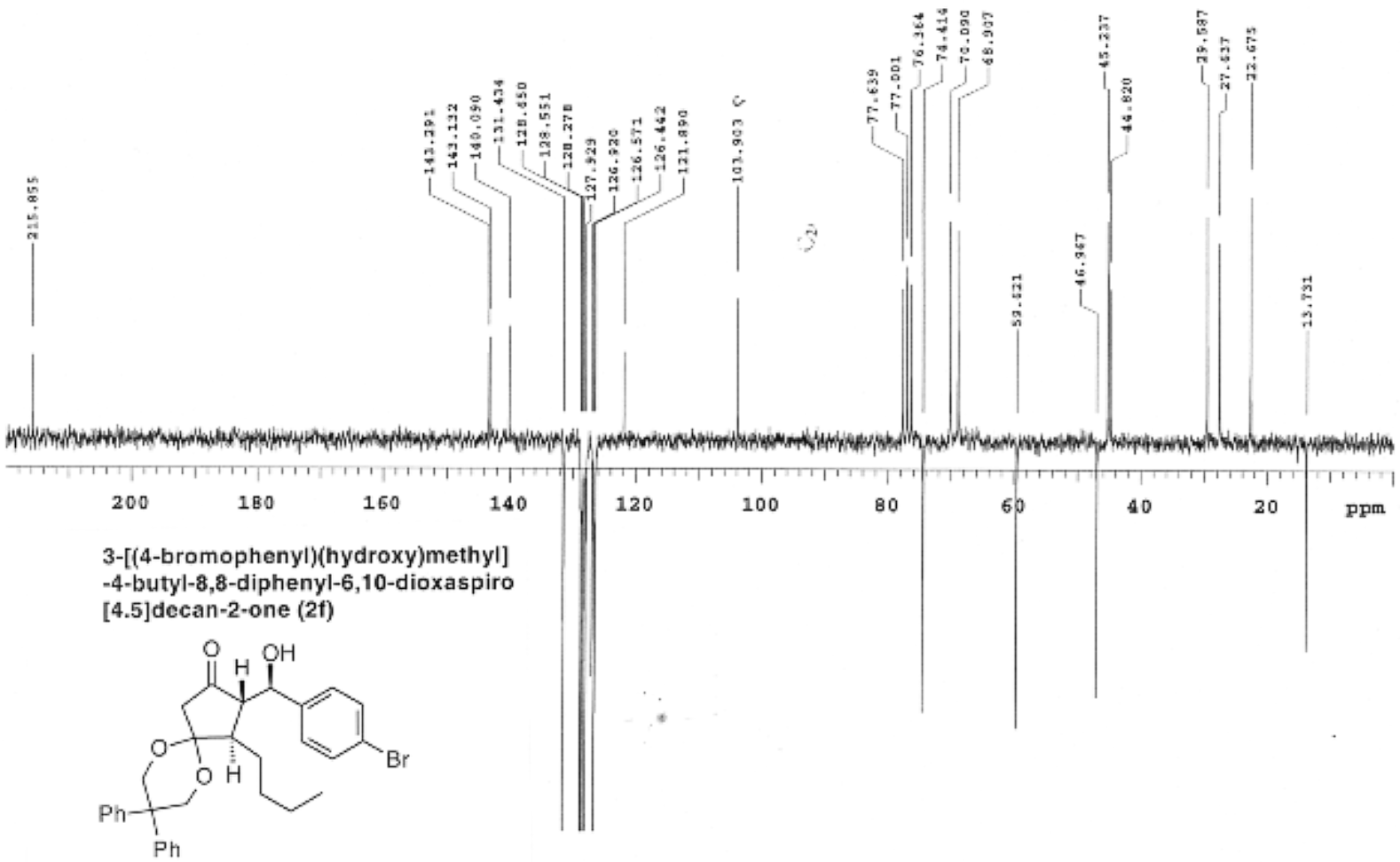
INDEX	FREQUENCY	PPM	HEIGHT
1	15332.909	203.267	14.7
2	11803.641	156.480	41.3
3	10819.694	143.436	18.6
4	10803.824	143.225	23.3
5	10193.435	135.133	40.4
6	9684.371	128.385	120.9
7	9669.722	128.190	107.6
8	9623.332	127.576	118.5
9	9557.410	126.702	126.0
10	9554.969	126.669	66.9
11	9534.216	126.394	50.1
12	7848.322	104.044	24.2
13	5840.142	77.422	5.6
14	5808.402	77.001	5.3
15	5775.441	76.564	5.5
16	5298.117	70.237	89.1
17	3348.535	44.391	22.4
18	3319.237	44.003	39.0

8,8,diphenyl-6,10-dioxa-spiro [4.5]dec-3-en-2-one (1b)

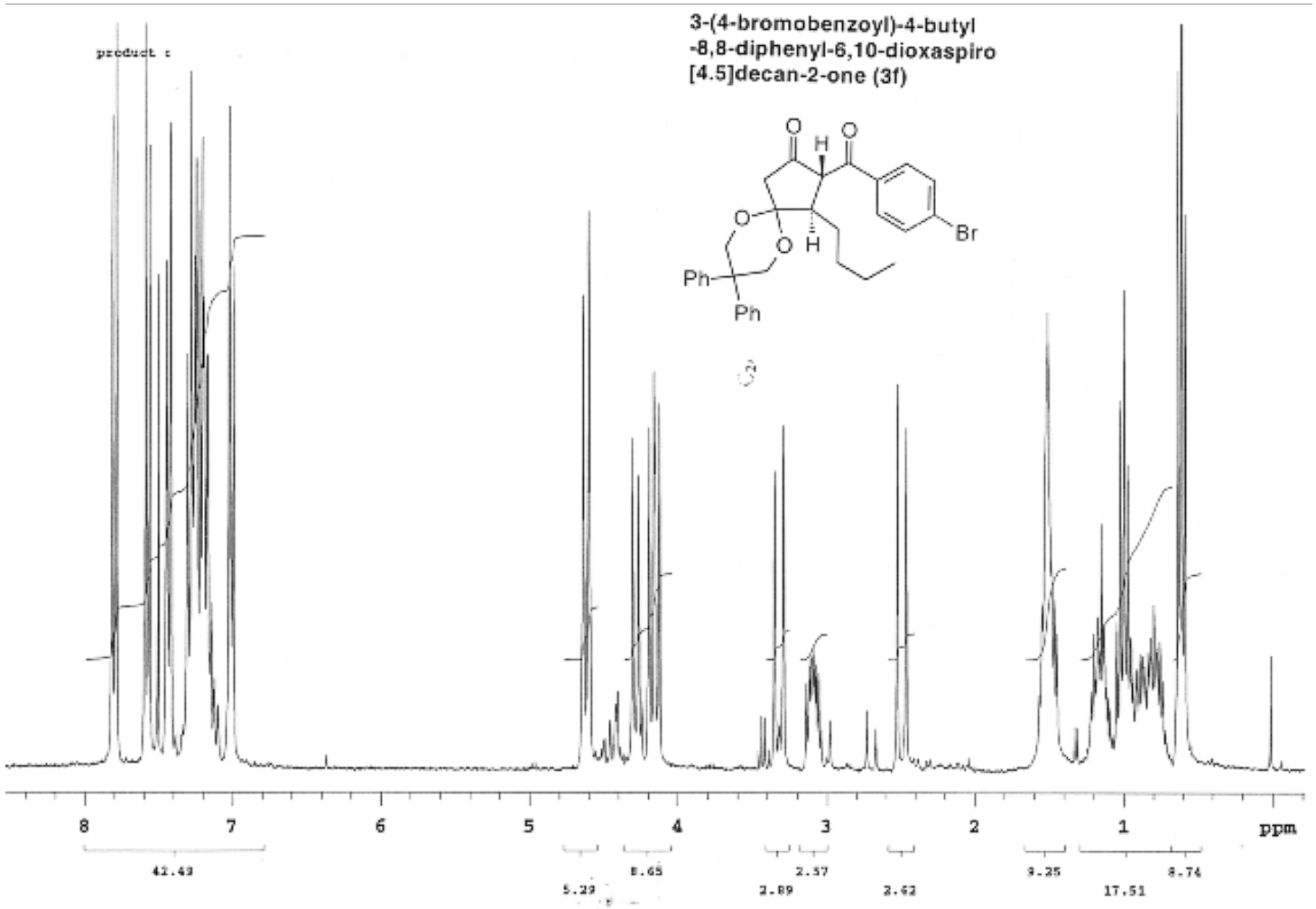


3-[(4-bromophenyl)(hydroxy)methyl]
-4-butyl-8,8-diphenyl-6,10-dioxaspiro
[4.5]decan-2-one (2f)





3-[(4-bromophenyl)(hydroxy)methyl]-4-butyl-8,8-diphenyl-6,10-dioxaspiro[4.5]decan-2-one (2f)


product :
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-8,8-diphenyl-6,10-dioxaspiro
[4.5]decan-2-one (3f)
O
H
O
O
O
H
Br
Ph
Ph
8
7
6
5
4
3
2
1
ppm
42.43
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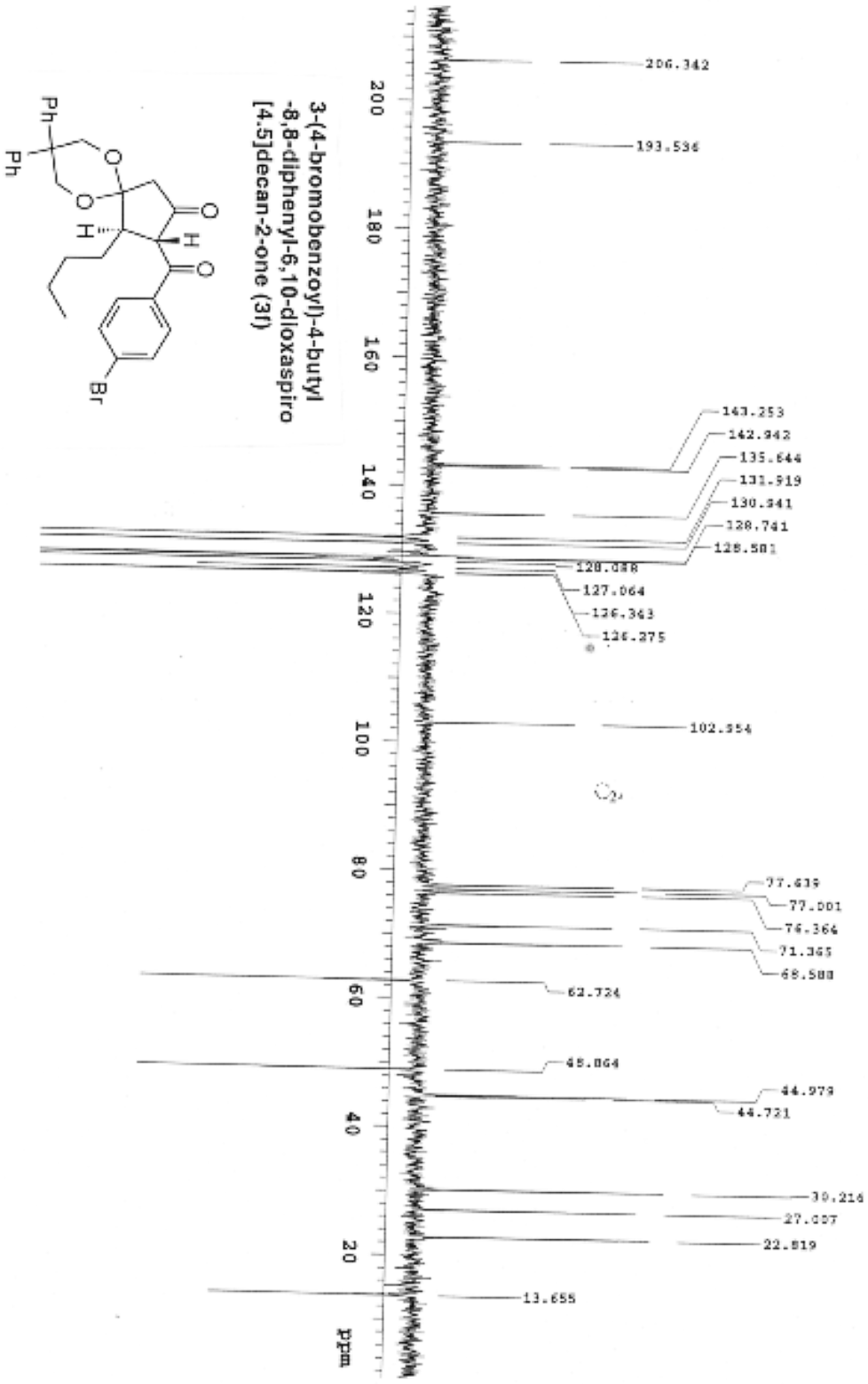
2.89
2.57
2.62
9.25
17.51
8.74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-8,8-diphenyl-6,10-dioxaspiro
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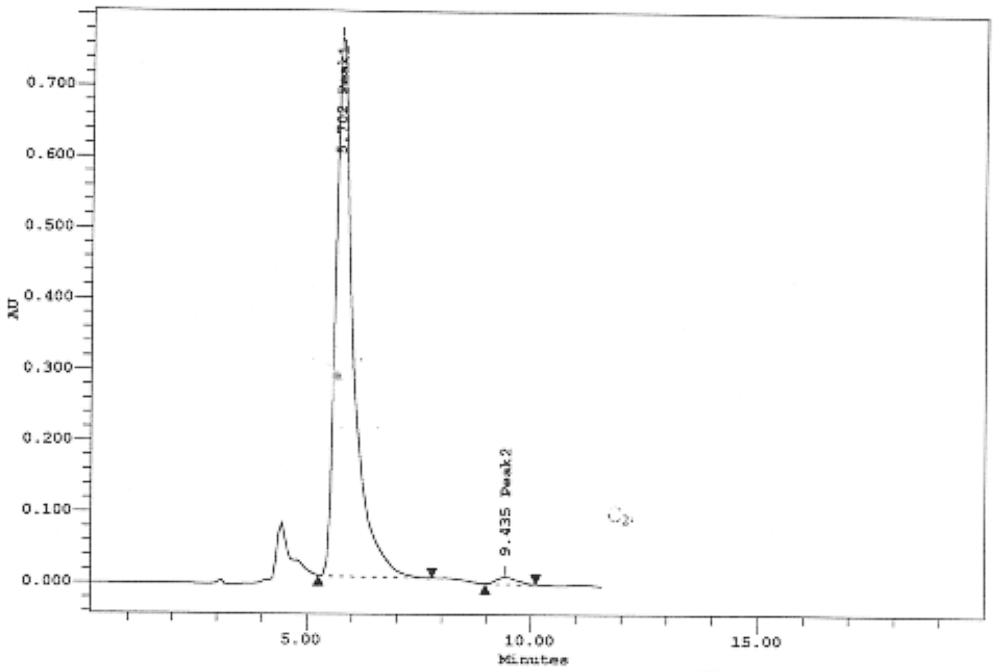
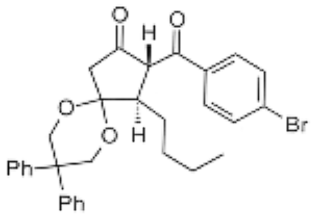
206.342
193.536
143.253
142.942
135.644
131.919
130.341
128.741
128.581
128.088
127.064
126.363
126.275
102.354
77.639
77.001
76.364
71.365
68.588
62.724
48.864
44.979
44.721
30.216
27.007
22.819
13.655
ppm

SampleName: Test Inject
Method Name: Leggy_rep
UserName: username
Acq Method Set: keller
Date Processed: 11/10/99 06:49:17
Current Date: October 11, 1999
Current Time: 06:50:45

Solvent: Solvent
Flowrate: flowrate
Column type: column type
Injection volume: 3.00 ul
Concentration: 1.00000
Det. Units: AU
Run Time: 20.0 min

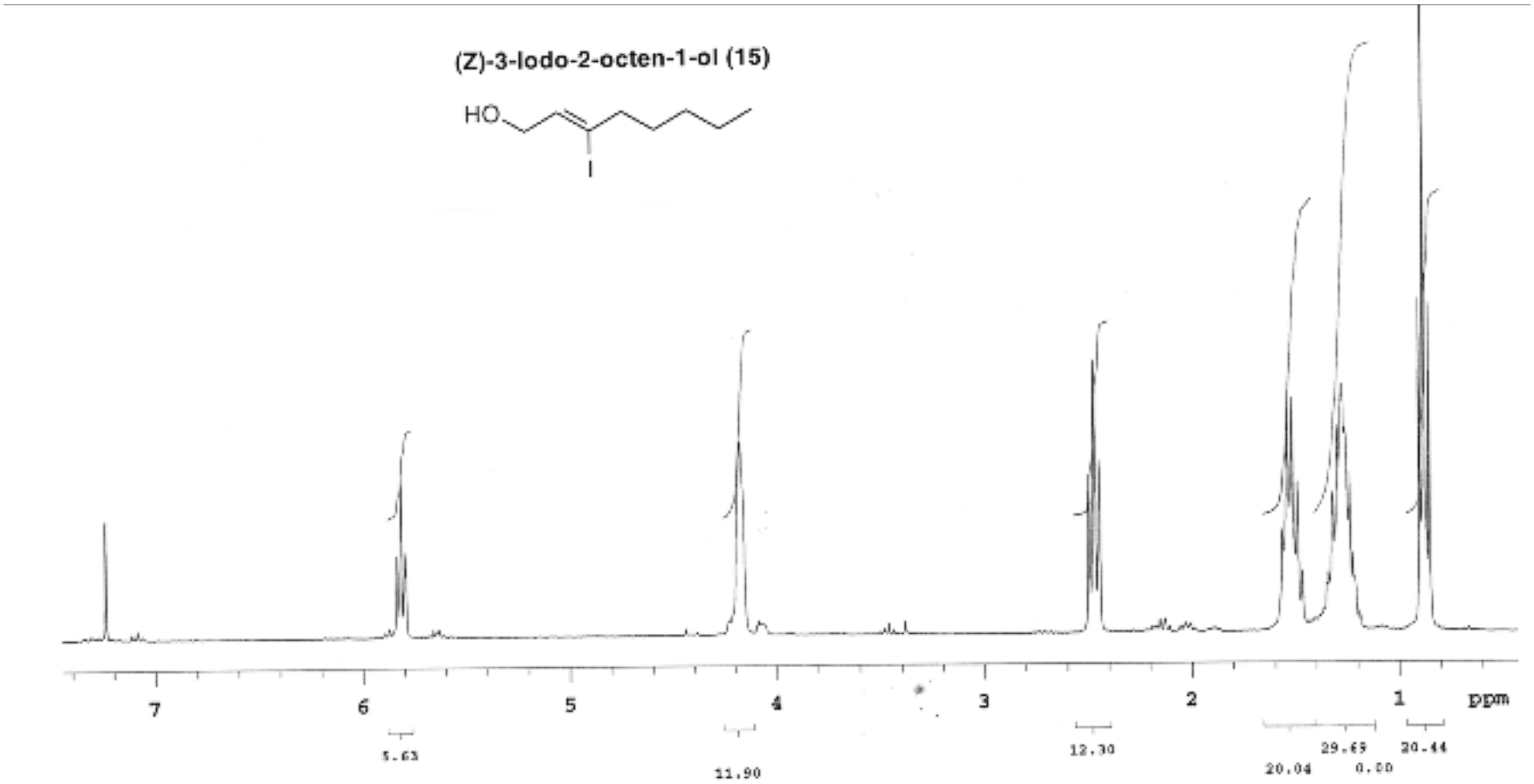


3-(4-bromobenzoyl)-4-butyl-8,8-diphenyl-6,10-dioxaspiro[4.5]decan-2-one (3f)

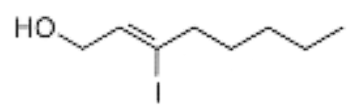


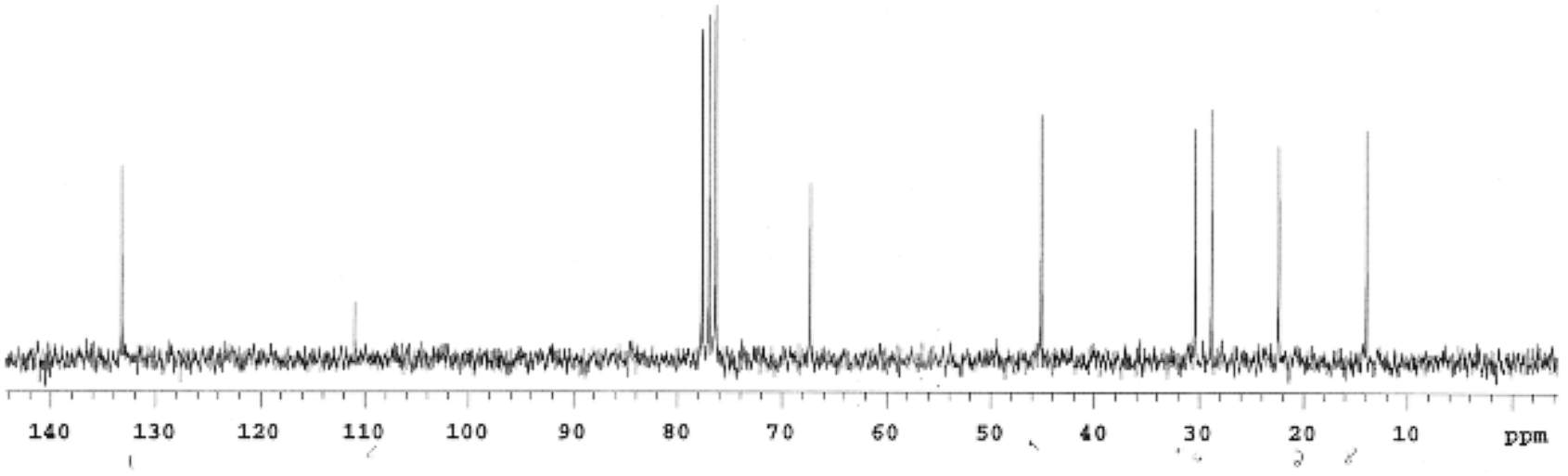
Peak Results

#	Name	Ret Time (min)	Area (uV*sec)	% Area	Height (uV)	Int Type
1	Peak1	5.702	20096030	98.58	756788	MM
2	Peak2	9.435	299968	1.42	10005	MM

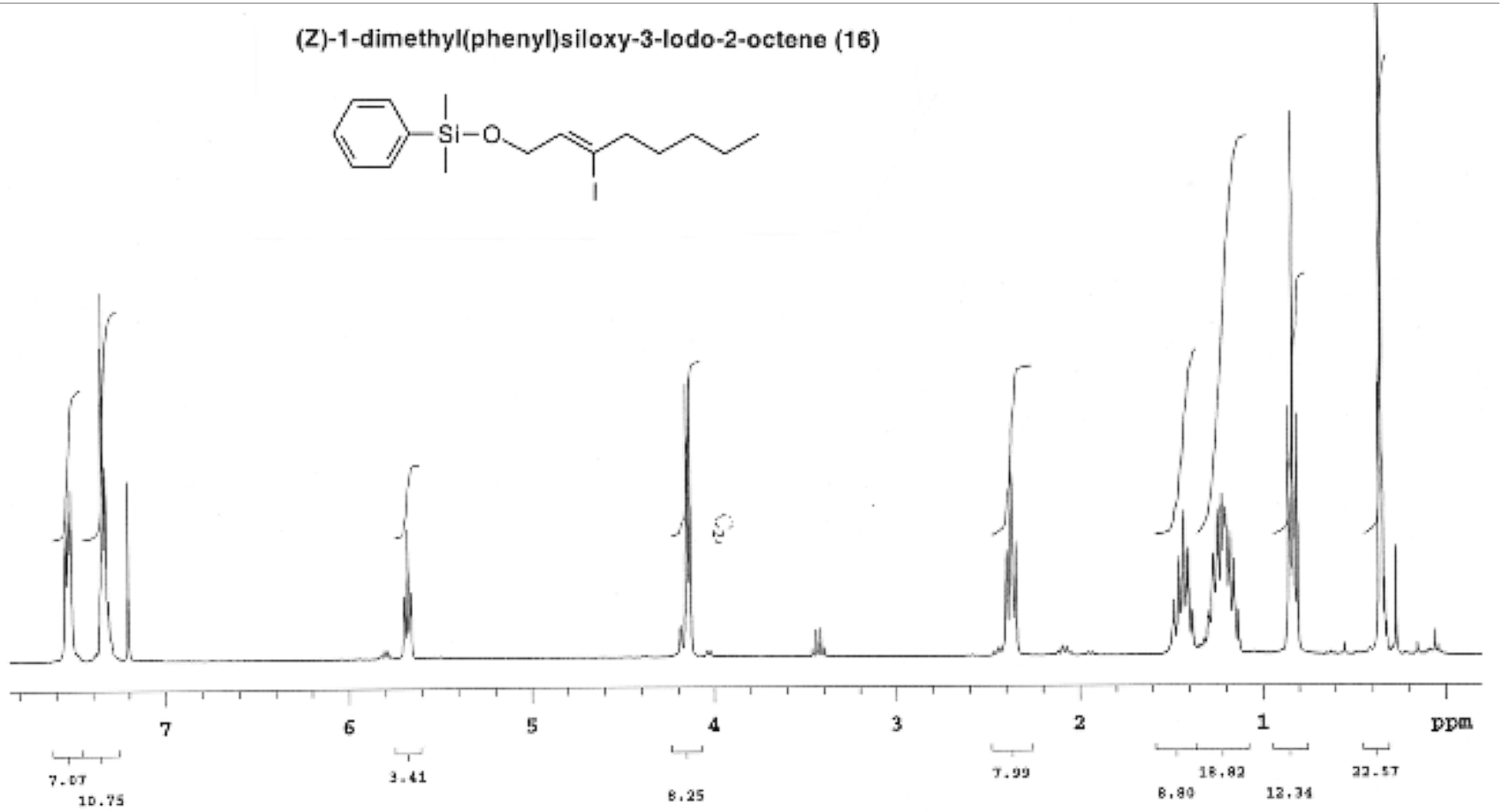

(Z)-3-Iodo-2-octen-1-ol (15)
HO
I
7
6
5
4
3
2
1
ppm
5.63
11.90
12.30
29.69
20.04
0.00
20.44

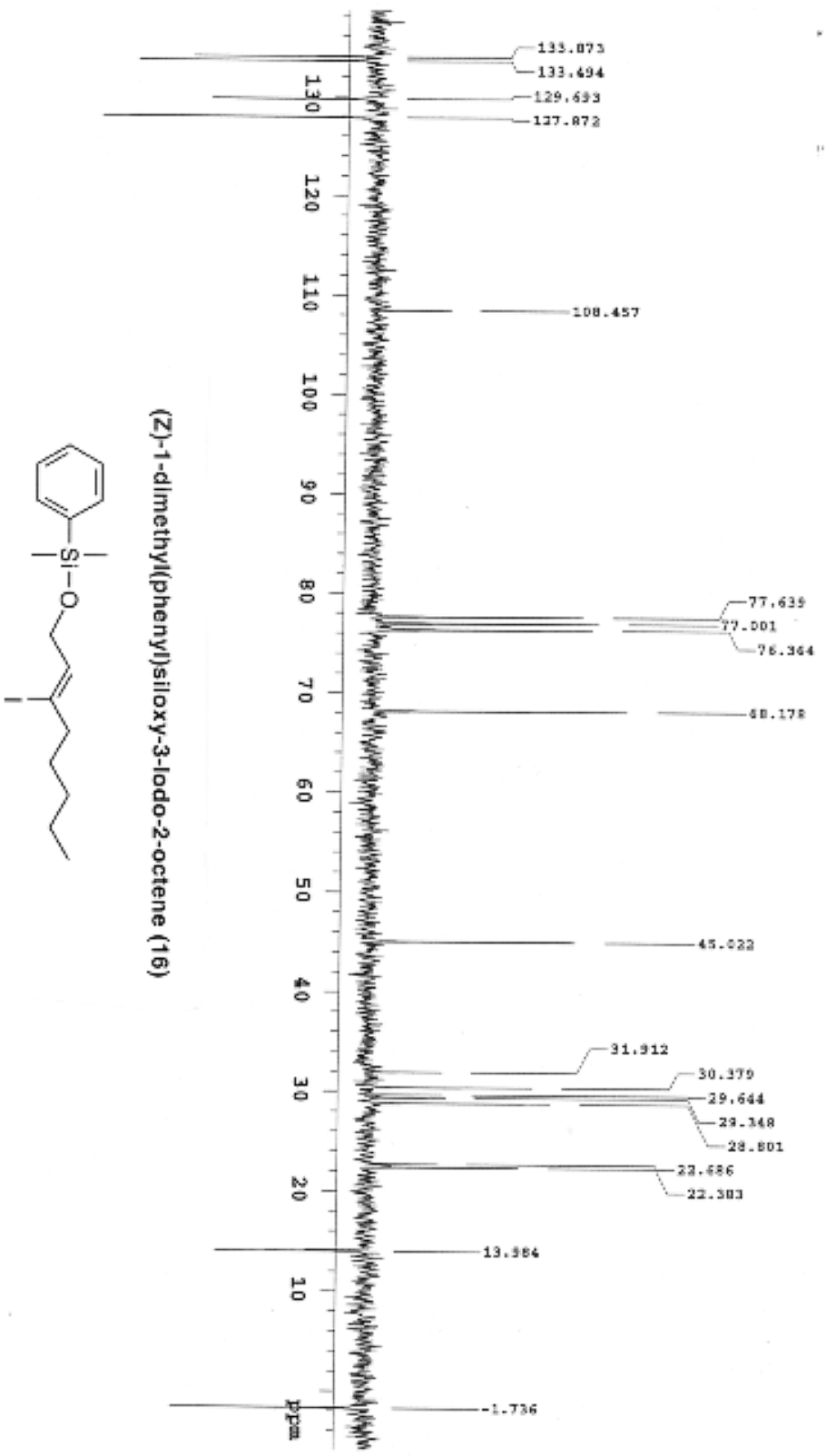
(Z)-3-Iodo-2-octen-1-ol (15)





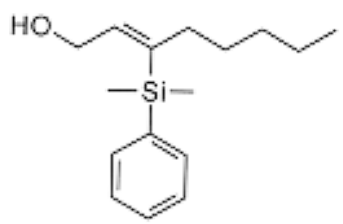
(Z)-1-dimethyl(phenyl)siloxy-3-Iodo-2-octene (16)



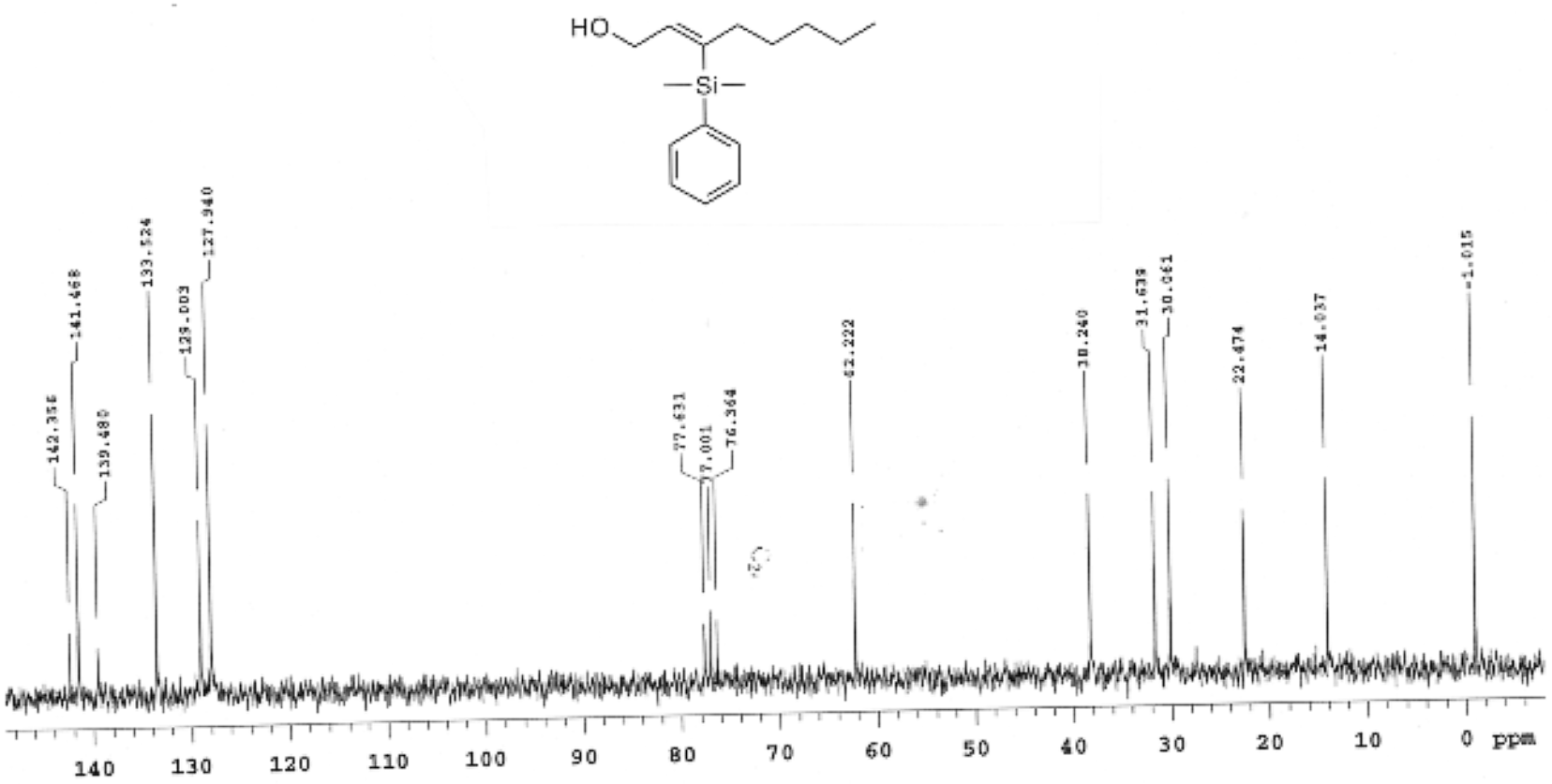


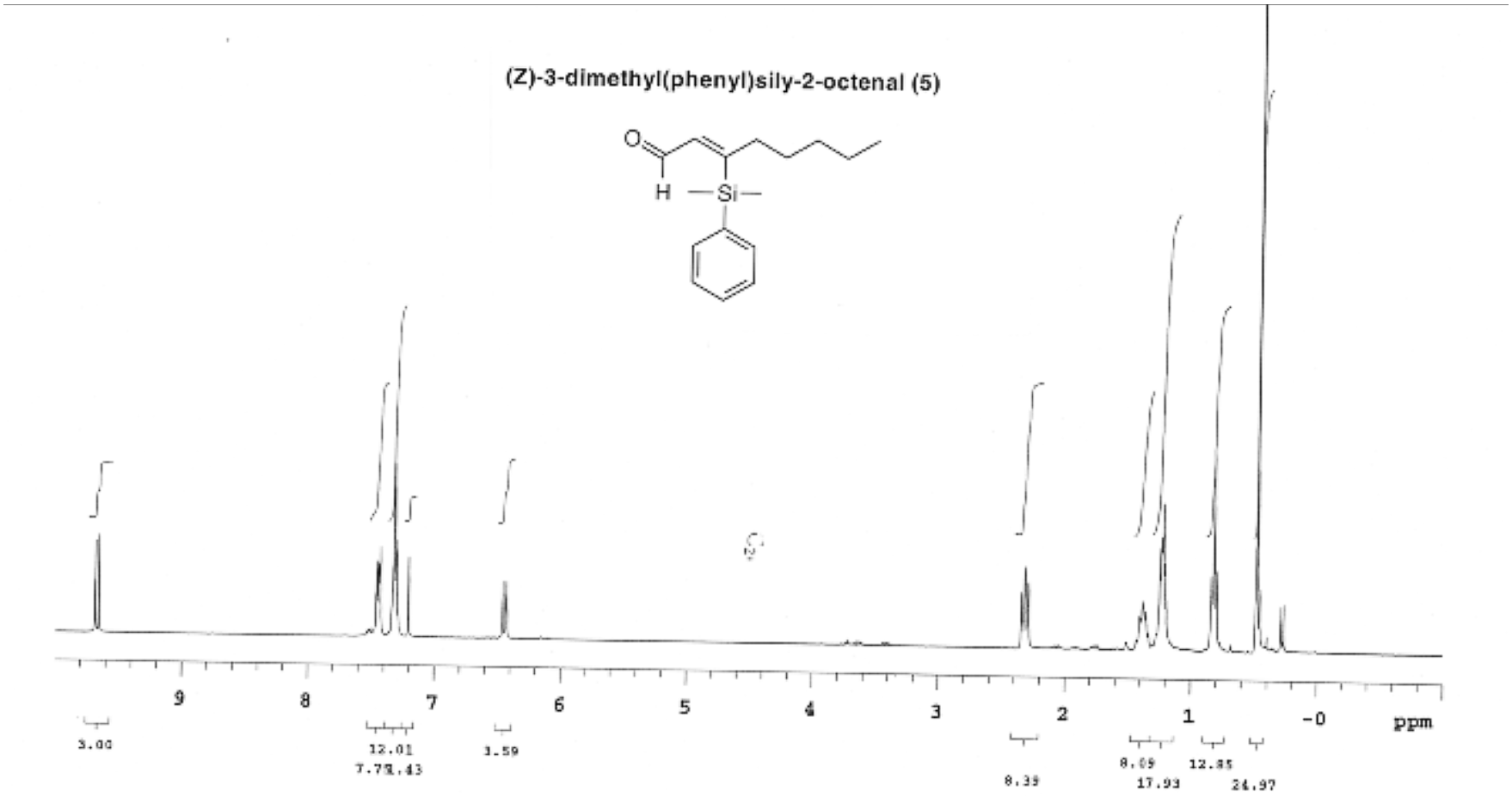
(Z)-1-dimethyl(phenyl)siloxy-3-Iodo-2-octene (16)

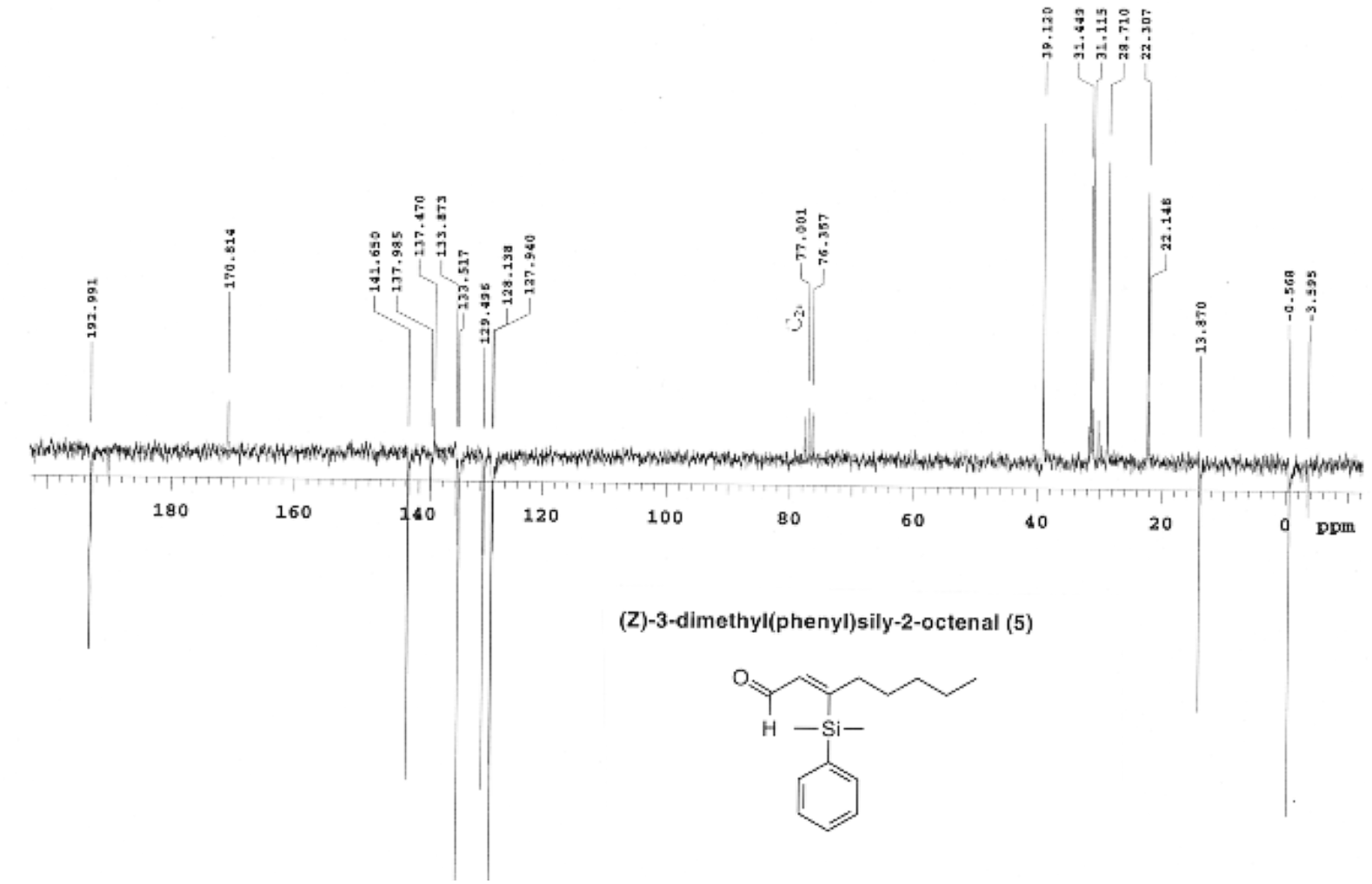
(Z)-3-dimethyl(phenyl)silyl-2-octen-1-ol (17).

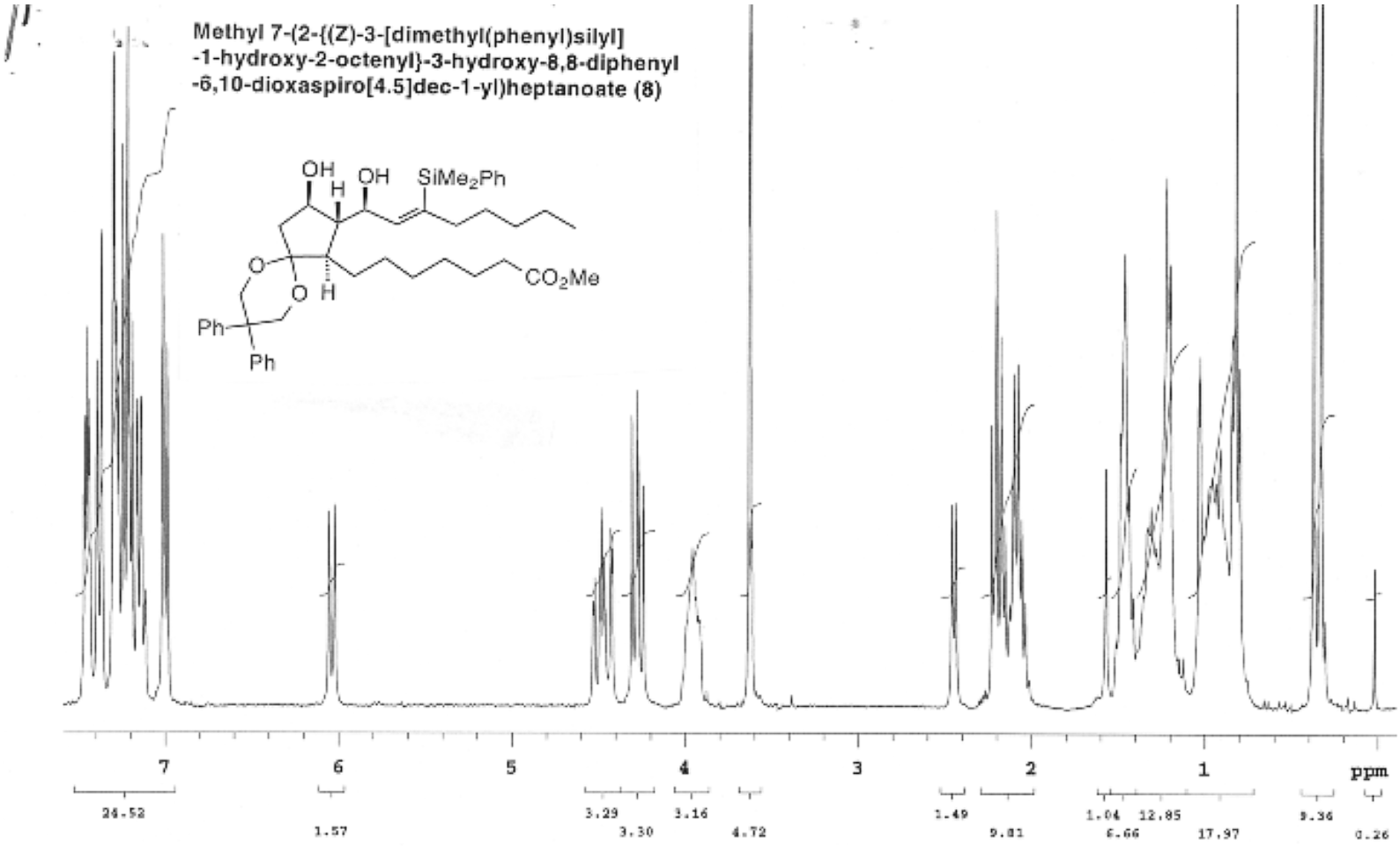


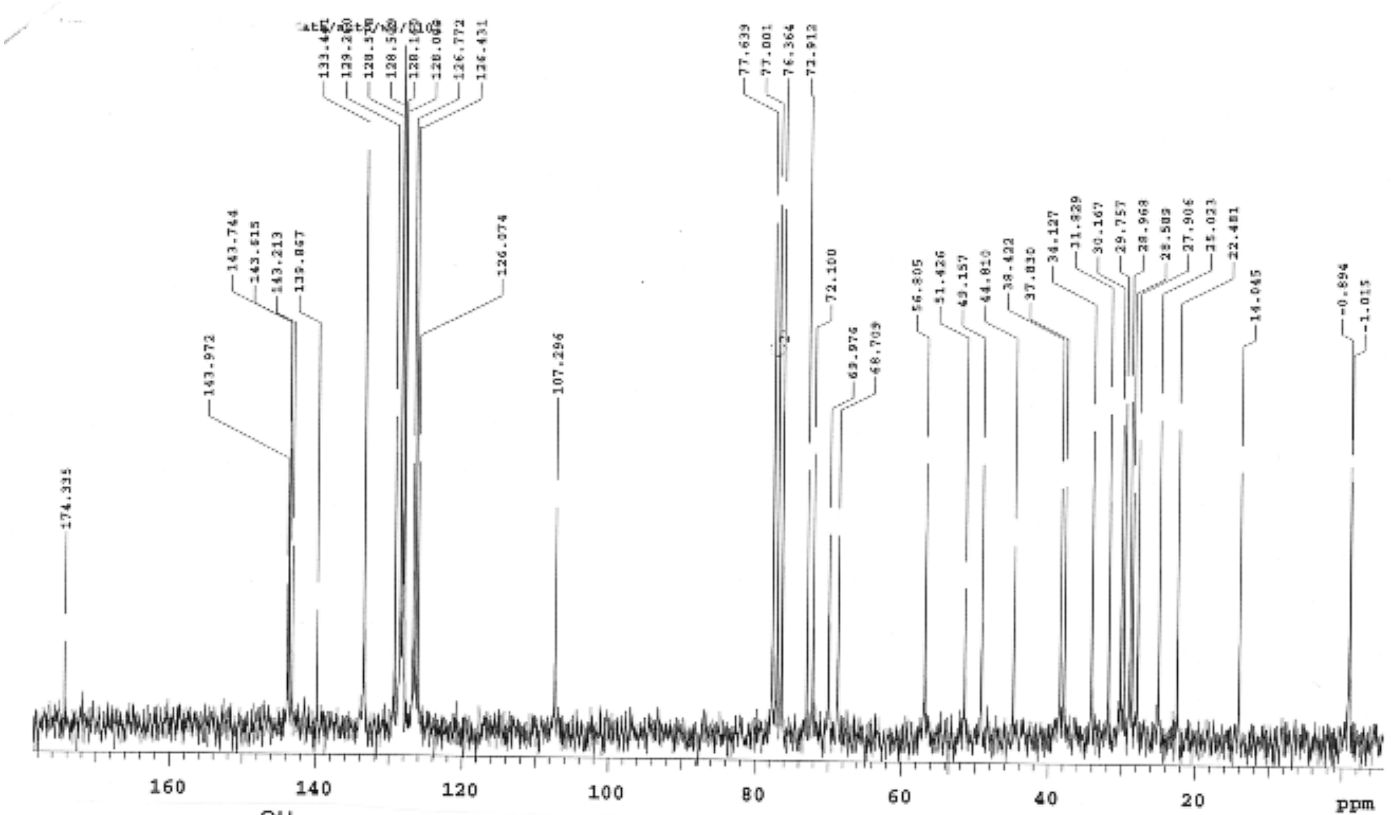
(Z)-3-dimethyl(phenyl)silyl-2-octen-1-ol (17).

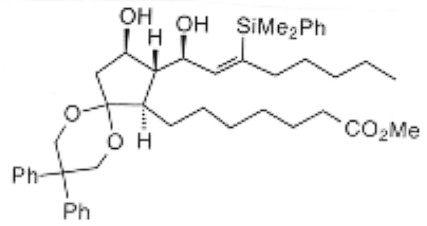



(Z)-3-dimethyl(phenyl)sily-2-octenal (5)
O
H
Si
9
8
7
6
5
4
3
2
1
-0
ppm
3.00
12.01
7.75
1.43
3.59
8.39
8.09
17.93
12.85
24.97


192.991
170.814
141.650
137.985
137.470
133.873
133.517
129.496
128.138
127.940
77.001
76.357
39.120
31.443
31.115
28.710
22.307
22.148
13.870
-0.568
-3.595
180
160
140
120
100
80
60
40
20
0
ppm
(Z)-3-dimethyl(phenyl)sily-2-octenal (5)
O
H
Si


Methyl 7-(2-{(Z)-3-[dimethyl(phenyl)silyl]
-1-hydroxy-2-octenyl}-3-hydroxy-8,8-diphenyl
-6,10-dioxaspiro[4.5]dec-1-yl)heptanoate (8)
OH
H
OH
SiMe2Ph
O
O
H
CO2Me
Ph
Ph
7
6
5
4
3
2
1
ppm
24.52
1.57
3.29
3.30
3.16
4.72
1.49
9.81
1.04
6.66
12.85
17.97
9.36
6.26



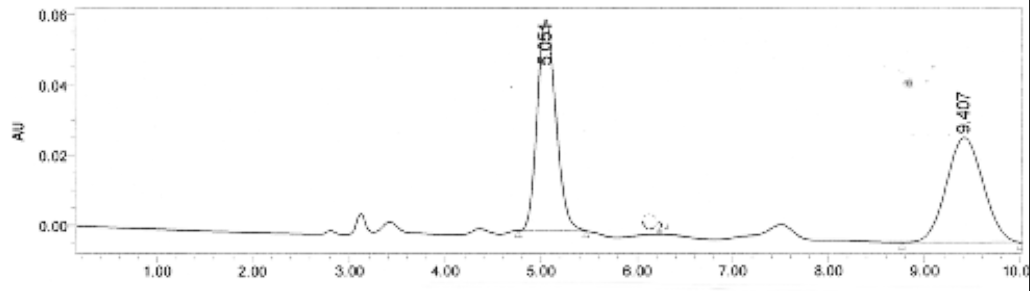


Methyl 7-(2-{(Z)-3-[dimethyl(phenyl)silyl]
-1-hydroxy-2-octenyl}-3-hydroxy-8,8-diphenyl
-6,10-dioxaspiro[4.5]dec-1-yl)heptanoate (8)

SAMPLE INFORMATION

Sample Name:	rac.	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	1/2/01 11:24:36 AM
Vial:	1	Acq. Method Set:	prostleggy
Injection #:	1	Date Processed:	1/2/01 12:11:26 PM
Injection Volume:	10.00 ul	Processing Method:	porstleggy
Run Time:	10.0 Minutes	Channel Name:	Wvin Ch1
Sample Set Name:		Proc. Chnl. Descr.:	PDA 261.2 nm

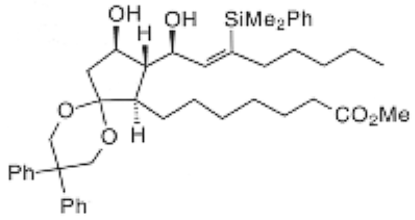
Sample structure:



	RT	Area	% Area	Height
1	5.051	808003	50.35	60011
	9.407	796785	49.65	29821

Methyl 7-(2-{(Z)-3-[dimethyl(phenyl)silyl]-1-hydroxy-2-octenyl}-3-hydroxy-8,8-diphenyl-6,10-dioxaspiro[4.5]dec-1-yl)heptanoate (8)

racemic material



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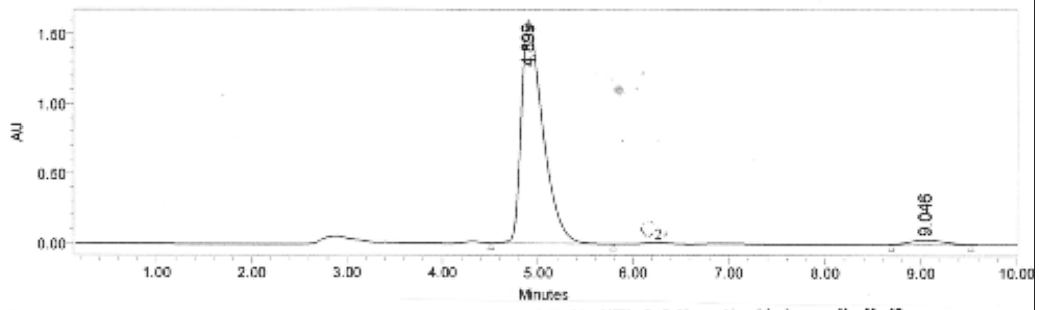
SAMPLE INFORMATION

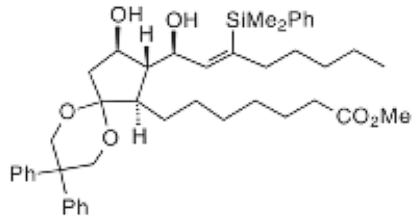
Sample Name:	~~rac~~ optical pure	Acquired By:	System
Sample Type:	Unknown	Date Acquired:	1/2/01 12:04:06 PM
Vial:	1	Acq. Method Set:	prostleggy
Injection #:	3	Date Processed:	1/2/01 12:27:49 PM
Injection Volume:	10.00 ul	Processing Method:	prostleggy
Run Time:	10.0 Minutes	Channel Name:	WvIn Ch1
Sample Set Name:		Proc. Chnl. Descr.:	PDA 262.3 nm

Sample structure:



	RT	Area	% Area	Height
1	4.899	24942833	96.82	1603449
	9.046	819096	3.18	33556

Methyl 7-(2-{(Z)-3-[dimethyl(phenyl)silyl]-1-hydroxy-2-octenyl}-3-hydroxy-8,8-diphenyl-6,10-dioxaspiro[4.5]dec-1-yl)heptanoate (8)



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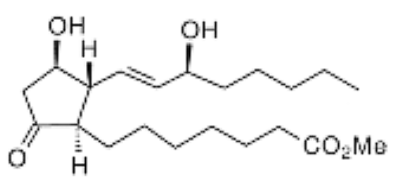
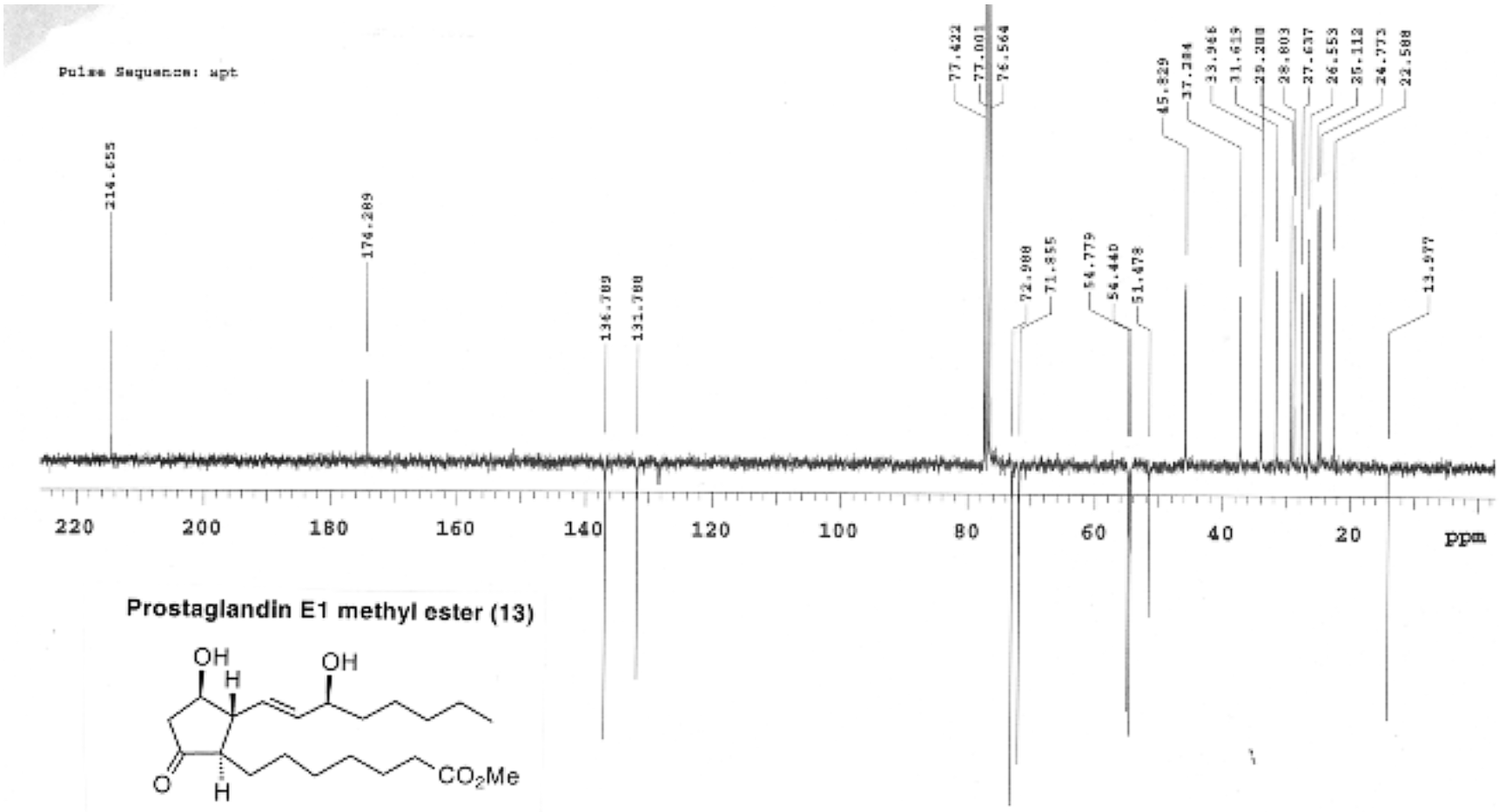
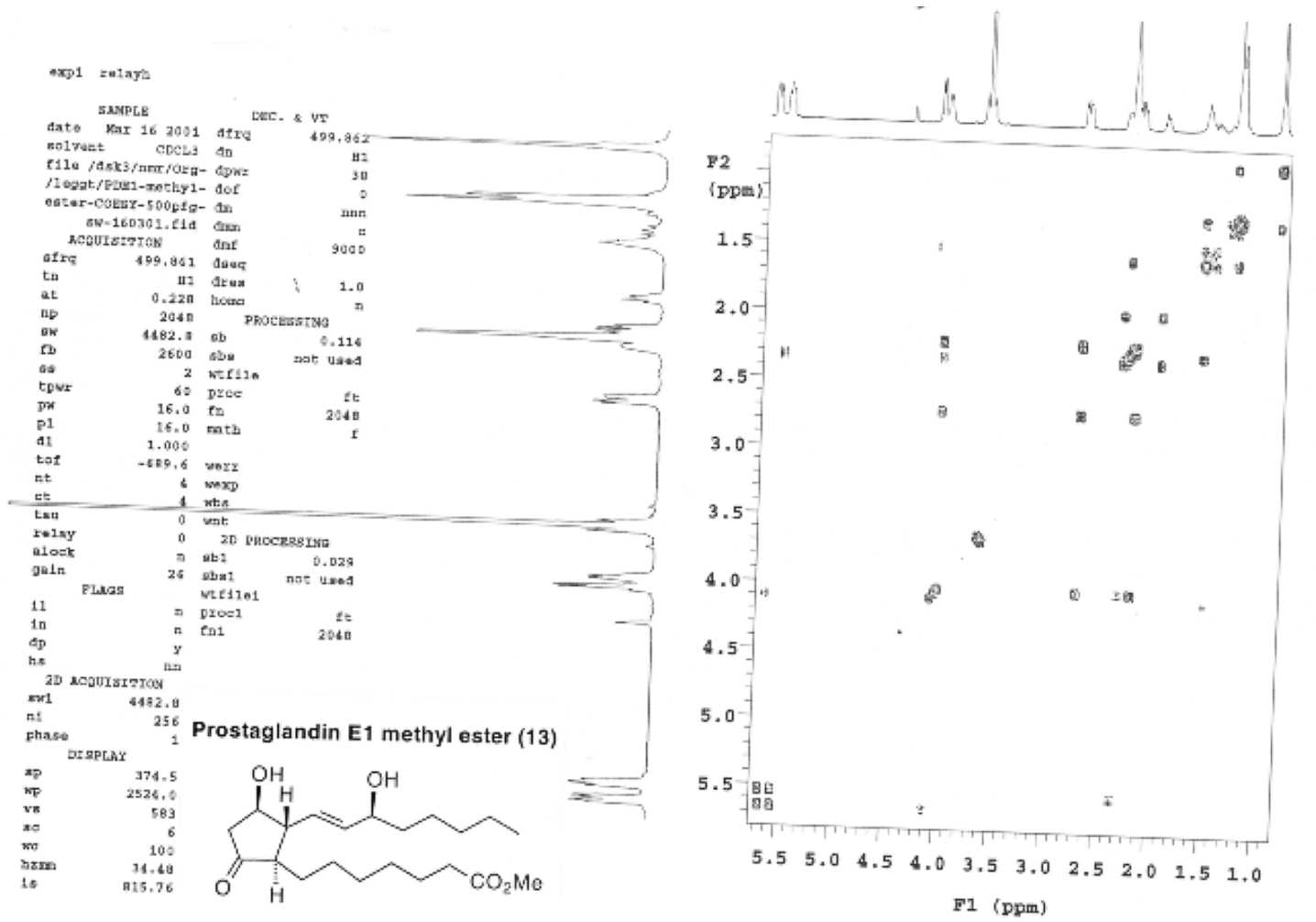
Prostaglandin E1 methyl ester (13)

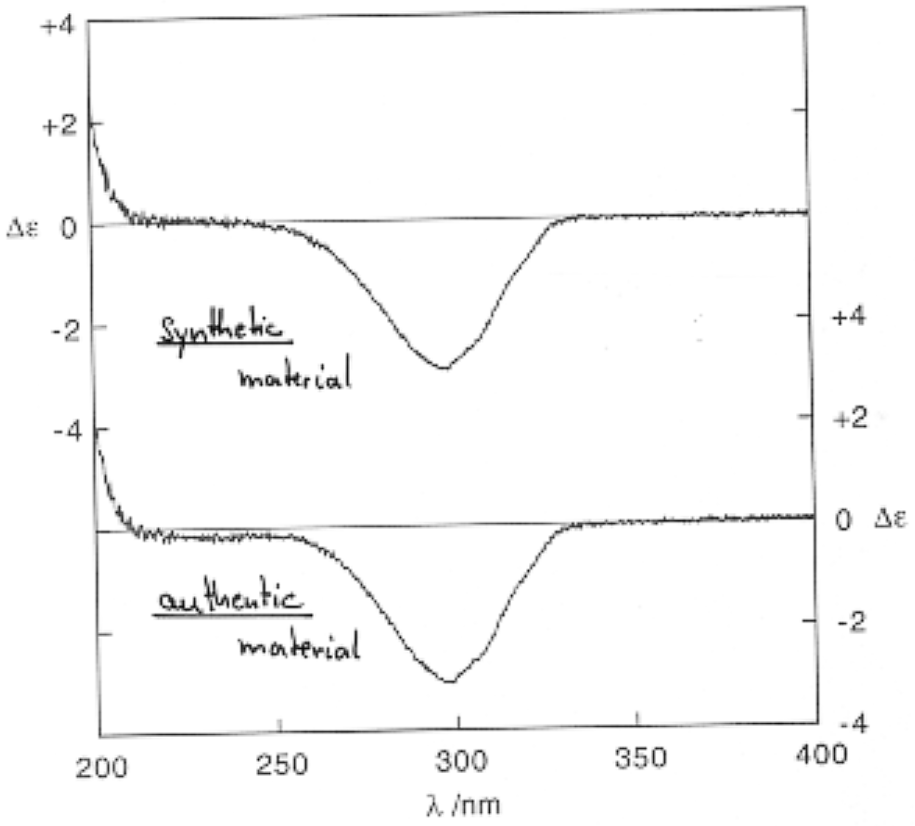




Prostaglandin E1 methyl ester (13)


expl relayh
SAMPLE
date Mar 16 2001
solvent CDCL3
file /dsk3/nmr/Org-/leggt/PDM1-methyl-ester-COSEY-500pfg-SW-160301.fid
ACQUISITION
sfrq 499.861
tn H1
at 0.228
np 2048
sw 4482.8
fb 2600
ss 2
tpwr 60
pw 16.0
pl 16.0
dl 1.000
tof -889.6
nt 6
ct 4
lau 0
relay 0
alock n
gain 26
FLAGS
il n
in n
dp y
hs nn
2D ACQUISITION
sw1 4482.8
ni 256
phase 1
DISPLAY
sp 374.5
wp 2524.6
vs 583
sc 6
wc 100
hzmm 34.48
is 815.76
DEC. & VT
dfrq 499.862
dn H1
dpwr 38
dof 0
dm nnn
dmm c
dmf 9000
dseq
drea 1.0
homo n
PROCESSING
sb 0.114
sbs not used
wtfile
proc ft
fn 2048
math f
werr
wexp
wbs
wnt
2D PROCESSING
sb1 0.029
sbs1 not used
wtfile1
proc1 ft
fn1 2048
Prostaglandin E1 methyl ester (13)
OH
H
OH
O
H
CO2Me
F2 (ppm)
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0
F1 (ppm)

comparison



Prostaglandin E1 methyl ester (13)